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# UNITED STATES DEPARTMENT OF AGRICULTURE



## DEPARTMENT BULLETIN No. 1371



Washington, D. C.



January, 1926

## EFFECTIVENESS AGAINST THE SAN JOSE SCALE OF THE DRY SUBSTITUTES FOR LIQUID LIME-SULPHUR

By

W. S. ABBOTT, Entomologist, and JULIAN J. CULVER, Assistant Entomologist,  
Enforcement of the Insecticide Act, Bureau of Entomology, and

W. J. MORGAN, Associate Chemist, Insecticide and Fungicide Laboratory,  
Miscellaneous Division, Bureau of Chemistry

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#### INTRODUCTION

Several dry substitutes for liquid lime-sulphur have been on the market for a number of years, but their value as remedies against the San Jose scale (*Aspidiotus perniciosus* Comstock) appears to be a disputed point. A brief abstract of the published experiments covering this point is given below.

The Arkansas station in 1921 (4, p. 18)<sup>1</sup> summarizes its work with certain sprays against the San Jose scale by stating that barium-sulphur,<sup>2</sup> calcium-sulphur, and sodium-sulphur "while highly effective in the season of 1921, were not so satisfactory during the two previous years, and can not yet be given as strong a recommendation as lime-sulphur."

In the annual report of the State entomologist of Colorado for 1922 (25, p. 47) experiments with a dry calcium-sulphur used on plots 6, 7, and 8, respectively, at the rate of 49, 50, and 56 pounds to 200 gallons of water are summarized as follows:

In the dry lime-sulphur plots, plot No. 8 shows a 3.08 per cent better kill than plot No. 6. This difference might possibly be attributed to the nozzle holder as different men did the work. Plot No. 7 shows perfect control, but the infestation was so light that a satisfactory count could not be made.

The Connecticut station in 1923 (5, p. 330) reports the following results of experiments against the San Jose scale: Liquid lime-sulphur, 77.3 per cent killed; barium-sulphur, 72.6 per cent killed; calcium-sulphur, 74.9 per cent killed.

<sup>1</sup> Reference is made by number (roman) to "Literature cited," pp. 25 and 26.

<sup>2</sup> For convenience in discussion the substitutes for liquid lime-sulphur are referred to as calcium, sodium, and barium sulphurs.

The Idaho station in 1918 (9, p. 9) says, "Dry lime-sulphur manufactured by the \* \* \* company was added to the experiment in 1917. It gave very encouraging results, but we are not prepared at this time to make any recommendations regarding this spray."

On page 15 results with a sodium-sulphur are given as follows: 1915, 20 pounds to 100 gallons of water, 64 per cent killed; 1916, 25 pounds to 100 gallons of water, 82 per cent killed; 1917, 25 pounds to 100 gallons of water, 93 per cent killed.

In the summary it is stated that the sodium-sulphur "gave fairly good results in 1917, but we can not recommend its use over lime-sulphur."

The Illinois station in 1919 (6, p. 4) records tests made with a sodium-sulphur, a barium-sulphur, and two calcium-sulphurs, showing the following results: Sodium-sulphur, excellent control; barium-sulphur, fair to good control; calcium-sulphurs, excellent control.

In Bulletin XIII of the Division of Natural History Survey for November, 1920 (10, pp. 341-342), additional tests are reported for one orchard as follows: Sodium-sulphur, good control; barium-sulphur, good control; calcium-sulphur No. 1, good to excellent control; calcium-sulphur No. 2, good control; calcium-sulphur No. 3, poor to fair control.

For another orchard: Sodium-sulphur, fair to good control; barium-sulphur, fair to good control; calcium-sulphur No. 1, good control; calcium-sulphur No. 2, good control; calcium-sulphur No. 3, very poor control.

The general conclusions are:

The results of two years' work with these materials seem to show that some dry sulphur compounds, if used at sufficient strength, are effective in controlling the San Jose scale. From the results of the past season where \* \* \* dry lime-sulphur was used at a strength of  $12\frac{1}{2}$  pounds to 50 gallons of water, it is apparent that these materials should not be used at a less rate than 15 pounds to 50 gallons of water.

In 1922 (22, p. 2) it is stated that "the diluted spray must contain 15 pounds of sulphur in 50 gallons," and in 1924 (2, p. 8) the following recommendation is made: Each 50 gallons of dormant spray should contain \* \* \* or  $15^3$  to  $28^4$  pounds of dry lime-sulphur \* \* \*.

In its annual report for the year 1919 (7, p. 44) the Kentucky station says:

*Use of dry lime-sulphur in sprays.*—Four samples of dry lime-sulphur were submitted by the department of horticulture for analysis. It had been noted that the use of the particular samples did not give the results expected.

The Missouri station in 1920 (13, p. 7) states:

Some report good results with dry lime-sulphur used at the rate of about 1 pound to 4 gallons of water. However, the writer's experience leads him to believe that in their present form the brands of dry lime-sulphur will not control this pest as effectively as the better brands of lime-sulphur solution.

In 1922 (14, p. 62) are reported tests with three calcium-sulphurs, one barium-sulphur, and one sodium-sulphur used at the rate of 12 and 20 pounds to 50 gallons. In no case were the dry materials as effective as liquid lime-sulphur, and the following conclusions are drawn: "The above results show that it was practically impossible to eradicate San Jose scale from infested peach trees by means of

<sup>1</sup> Recommendation for points north of Hancock and Vermilion Counties.

<sup>2</sup> Recommendation for Hancock and Vermilion Counties and points in same latitude or south.

a spray, but the proper application of most of the materials listed gave good control."

In 1923 (*15*, p. 2) it is stated:

During the past three years most growers in this State have controlled the scale with lime-sulphur, though some have lost faith in it. Dry lime-sulphur preparations have failed in our experiments to give satisfactory control.

The New Mexico station in its annual report for 1918-19 (*12*, p. 15) says:

*5. San Jose scale.*—Dry lime-sulphur and concentrated lime-sulphur were used as sprays for the above scale. The dry lime-sulphur dissolves readily, with little or no residue, is much more easily prepared, saves time, and is just as effective. Many of the chemical spray companies think the prices will be so reduced in another year that every farmer and orchardist will be able to use it. Between the two above sprays, the dry lime-sulphur is the coming insecticide for the San José scale.

In the report of the New York station for 1923 (*20*, p. 41) is found the following statement:

In this season's efforts provision was made for tests of various commercial sulphides in a powdered or granular state in comparison with lime-sulphur solution at standard strength. On the basis of the initial killing of the San Jose scale on old apple trees, the dry sulphides were noticeably inferior to lime-sulphur, while oil emulsions gave better control of the pest than lime-sulphur.

In the monthly bulletin of the Ohio station for February, 1920 (*16*, pp. 50-51), tests are reported with one calcium-sulphur, one sodium-sulphur, and one barium-sulphur, and it is stated that "commercial or practical control" was obtained.

In a later issue (*1*, p. 25) the following statement is made:

The powdered lime-sulphur has not yet been sufficiently tested to warrant us in recommending it as a perfect substitute for the liquid lime-sulphur, but we have cleaned an orchard badly encrusted with San Jose scale by applying it in the spring, 12 pounds to 50 gallons of water. \* \* \* Until more extensively tested we do not recommend it as being so reliable for cleaning badly infested orchards as liquid lime-sulphur or miscible oil, but believe it can be safely used for a few seasons, at least, as a dormant spray in orchards only slightly or not at all infested with scale. For dormant use, mix 15 pounds with 50 gallons of water.

The Oregon station in 1924 (*21*, p. 8) states that barium-sulphur "has been shown to be a fairly effective contact poison, but it possesses no distinct advantage over the lime-sulphur solution except that it is in powder form and convenient to handle," and sodium-sulphur "is similar to lime-sulphur except that lye is used instead of lime in its preparation. As a dormant spray it has been found satisfactory, but it has no superiority over the lime-sulphur."

In reference to calcium-sulphur this bulletin says:

When dry lime-sulphur is used either for the dormant spray or for the summer sprays it should be applied in amounts equivalent to the liquid lime-sulphur in order to obtain equivalent protection. Since each gallon of lime-sulphur solution contains 3.4 pounds of active ingredients it would take 4 pounds of a dry lime-sulphur having 85 per cent active ingredients to be equivalent to 1 gallon of liquid lime-sulphur. Therefore, in making dilutions for the various sprays 4 pounds of the dry lime-sulphur should be used for each gallon of liquid lime-sulphur, Baumé 33°, necessary. While this amount is much higher than is recommended by the manufacturer, field experiments carried on in different parts of the country have indicated conclusively the need of using larger amounts than the manufacturers have recommended in the past, particularly when weather conditions favor the development of serious disease epidemics.

The Texas station in 1920 (*24*, p. 7) summarizes the results of tests made with a sodium and a calcium-sulphur and a commercial liquid lime-sulphur as follows:

The fact that infestation increased heavily on all the trees in the check rows while at the same time there was nearly perfect control in the case of the sprayed trees, shows that all three materials were very effective in controlling the San Jose scale under the conditions of this experiment. So far as we could determine, there was very little if any difference in the effectiveness of the three materials.

It is realized, of course, that this is only a preliminary experiment and that further tests should be made with such mixtures before final recommendations can be made.

Numerous experiments with dry sulphur preparations are reported by the Washington station (*17*; *18*, p. 21) and the conclusions are drawn that in certain parts of the State none of the sulphur sprays are effective against the San Jose scale.

In the April, 1924, issue of the *Journal of Economic Entomology* (*8*, p. 288-289), J. J. Davis, of Purdue University, reports a large series of tests made with various preparations against the San Jose scale, and says:

*Results.*—We have concluded from these tests and many scattered observations, that the dry lime-sulphur is inefficient against the San Jose scale as it occurs at the present time in southern Indiana when used at label strength. The liquid concentrate proved ineffective under the conditions which have prevailed in southern Indiana the past few years. These results are corroborated by results secured where the scale could not be checked even when 1-6 strengths were used thoroughly. Even with a 90 per cent kill, the 10 per cent live scales on moderately or heavily infested trees are able to increase and encrust a vigorous tree by fall. The dry lime-sulphur when used at twice label strength was about equal in effectiveness to the liquid concentrate.

The lack of agreement in the conclusions reached by the investigators quoted above may be explained in several ways:

(1) The results were taken in many different ways and, in some cases, the methods used apparently do not correctly represent the effect of the treatment on the hibernating scale.

(2) These experiments cover a period of six years, and it is generally recognized by entomologists that the virility of this scale in a given locality may vary greatly in the course of several years, and, moreover, some of these tests were made when the vitality of the scale was very low. This has been well shown in Arkansas (*3*) where, prior to 1918, the scale had been kept in check by one dormant treatment with liquid lime-sulphur. From 1919 to 1922 this pest became so virulent that liquid lime-sulphur could no longer be relied on, even when two applications at greatly increased strengths were used. That this was not the result of faulty spraying is shown by experiments of the Bureau of Entomology at Bentonville, Ark., in 1921 and 1922.

(3) It is also a well-established fact that the vitality of this species of scale varies with the locality, and some of these experiments were made in sections where the scale is not difficult to control. This regional variation in resistance has been demonstrated by the work of A. L. Melander (*19*) in Washington.

(4) In many cases only the percentage of dead or living scale is given and such results are very misleading if the number of dead scales in the untreated plats, or checks, is not taken into consideration.<sup>5</sup>

<sup>5</sup> See "Percentage of control," p. 6.

In view of these very conflicting data, further experiments were deemed necessary, and the entomologists of the Insecticide and Fungicide Board, working under the direction of the Bureau of Entomology, have, therefore, very carefully tested a representative series of the dry substitutes for liquid lime-sulphur, as dormant sprays against the San Jose scale.<sup>6</sup>

These preparations have been tried at several dilutions under practical orchard conditions as dormant sprays against the San Jose scale on peach trees in Mississippi and Alabama and on apple trees in Indiana and Virginia. In every case unsprayed trees and trees sprayed with liquid lime-sulphur were included as controls.

The tests, which cover a period of three years and have been carried on in four different States, form the basis of this report and they are confirmed by numerous other experiments that have been made in cooperation with the writers in other parts of the country.<sup>7</sup>

### MATERIALS USED

All of the materials used in these experiments were purchased in the open market, analyzed, and kept under seal until tested. New samples were collected each year and nothing but fresh material, which had not been exposed to the air, was used. Although the different samples vary slightly in chemical composition, they are, on the whole, very similar and typical of the dry substitutes for liquid lime-sulphur now being sold. The analyses of the preparations tested are given in tables preceding each set of experiments.

### LIQUID LIME-SULPHUR

A good commercial liquid lime-sulphur solution should test from 32 to 33° Baumé (sp. gr. 1.283 to 1.295), and analyze approximately as follows: Calcium polysulphide 30 to 32 per cent; calcium thiosulphate 1.5 to 2.5 per cent; traces of calcium sulphate; impurities from the lime; and the remainder water. A comparison of the composition of the liquid and dry lime-sulphurs shows that the latter contain roughly about twice as much calcium polysulphides; from three to five times as much calcium thiosulphate; and, in addition, from 8 to 14 per cent of free sulphur, which does not occur in liquid lime-sulphur solution.

Since a gallon of commercial liquid lime-sulphur (32° Baumé) weighs about 10.7 pounds and contains approximately 32 per cent of calcium polysulphides, a dilution of 6.66 gallons to 50 gallons of spray (1 to 7.5), which was the strength used in the tests here reported, will furnish approximately 22.8 pounds of calcium polysulphides in each 50 gallons of spray material.

### RECORDS TAKEN

Two kinds of records were taken in these experiments, one on the hibernating scales and one on the young scales settling on new wood.

In order to determine the effect of the treatment on the hibernating scales, a number of twigs and branches were cut from all parts of the

<sup>6</sup> The field experiments were under the direct supervision of J. J. Culver, who also made nearly all of the records.

<sup>7</sup> A. J. Ackerman in Arkansas, O. I. Snapp in Mississippi, J. J. Culver in Georgia, and J. E. Fouser and J. J. Davis in Indiana.

record trees. These were placed under a binocular, the scale covering was lifted with a needle, and the scale examined to determine whether it was living or dead. Two thousand scales were counted from each plat, except from those used in the 1921 experiments in Indiana, where only 1,000 were counted. In this examination only the hibernating females were recorded, the last season's dead scales and the young scales, which would not in any case survive the winter, being disregarded. These counts were made approximately one month after treatment.<sup>8</sup>

In addition to the examination of the hibernating scale to determine the percentage actually killed by the spray, a careful count of the newly settled scale on 80 linear inches of new wood was made. This was done for the first and second generations on peach trees and for the first generation on apple. These records show the continued effect of the dormant spray as well as the value of the treatment in preventing reinfestation of the treated trees.

For this purpose a number of twigs with a total length of 80 linear inches were selected from each plat, care being taken to secure representative twigs from branches with approximately the same original infestation. The number of newly settled scales on these 80 inches of wood was counted and the average number per inch taken as a measure of the reinfestation resulting from the scale not killed by the dormant spray.

#### PERCENTAGE OF CONTROL

In computing the actual value of a given treatment against an insect where it is possible to make an accurate count of the living and dead individuals and no satisfactory data can be obtained to show the effect of the infestation on the host plant or the crop produced, it is necessary to take into account the number of dead insects in the untreated check. In the following experiments the figure designated "percentage of control" is obtained as follows:

Let  $X$  = per cent living in the untreated check.

Let  $Y$  = per cent living in the treated plat.

Then  $X - Y$  = per cent actually killed by the treatment.

The ratio of the percentage actually killed by the treatment ( $X - Y$ ) to the percentage living in the check ( $X$ ) will give the actual

efficiency of the spray, or  $\frac{X - Y}{X} \times 100$  = per cent control.

#### DRY CALCIUM-SULPHURS

These so-called dry lime-sulphurs, which are sold as substitutes for liquid lime-sulphur solutions, are of comparatively recent development, and their sale has reached large proportions. The great quantity of water in lime-sulphur solutions makes this product objectionable as a commercial preparation from the standpoint of packing and shipping. Many efforts have been made to eliminate this feature and to obtain a lime-sulphur product in dry form that is at the same time susceptible to ready solution. Several "dry" methods of preparation have been tried, but in general the method of manufacture

<sup>8</sup> A. L. Melander (19) has shown that an examination made at this time gives the most reliable index of the efficiency of a dormant spray against the San Jose scale.

is first to prepare commercial lime-sulphur solution in the usual manner, then to add a "stabilizing" substance (usually cane sugar), and finally to evaporate the solution to dryness, either in vacuo or at atmospheric pressure in the presence of an inert gas. Several patents have been issued for the preparation of a dry lime-sulphur,<sup>9</sup> but practically all of that produced at the present time is made by the process of evaporation under reduced pressure in the presence of a "stabilizer."

#### CHEMICAL COMPOSITION

Commercial dry calcium-sulphur, or dry lime-sulphur as it is usually called, contains a relatively large percentage of insoluble matter, consisting chiefly of free sulphur and smaller amounts of calcium sulphite and lime. This high percentage of insoluble matter is objectionable because it decreases the active ingredients and tends to clog the nozzle of the spraying apparatus. The average composition of six different brands of dry lime-sulphurs,<sup>10</sup> representing the principal brands on the market, are given in Table 1.

TABLE 1.—*The chemical composition of representative dry lime-sulphurs*

Brand	Average percentage found				Number of samples averaged
	Calcium polysulphides <sup>1</sup>	Calcium thiosulphate <sup>2</sup>	Free sulphur	Other ingredients (diff.) <sup>3</sup>	
A-----	67.31	6.96	8.44	17.29	10
B-----	64.67	9.66	10.31	15.36	10
C-----	70.59	8.72	8.09	12.60	4
D-----	59.68	8.15	11.79	20.38	5
E-----	60.82	6.42	14.08	18.68	4
F-----	64.60	9.09	8.87	17.35	10
Weighted average-----	64.90	8.34	9.86	16.91	43

<sup>1</sup> The sum of the polysulphide sulphur and the polysulphide calcium.

<sup>2</sup> Thiosulphate sulphur calculated to calcium thiosulphate,  $\text{CaS}_2\text{O}_3$ .

<sup>3</sup> Mainly water, sugars, calcium sulphite and insoluble impurities.

#### EXPERIMENTS IN 1921

For the experiments made in Indiana in 1921 an old apple orchard at Washington, Ind., badly infested with the San Jose scale, was used. This was divided into plats of three to five trees, each with approximately the same infestation. A dormant spray was applied on March 22 and 23, using a power sprayer maintaining a pressure of 225 pounds, with two spray guns. From 9 to 12 gallons of spray were applied to each tree.

The composition and dilutions of the essential materials used are given in Table 2.

<sup>9</sup> United States patents Nos. 460,227; 997,601; 1,186,564; 1,231,741; 1,254,908; 1,336,957; 1,338,678; 1,374,951; 1,422,977; and 1,423,605.

<sup>10</sup> Material purchased on the open market.

TABLE 2.—*Composition of the dry calcium-sulphurs tested in experiments against the San Jose scale in 1921*

Material used	Per cent of—			Quantity used in 50 gallons of spray	Quantity of poly-sulphides in 50 gallons
	Calcium polysulphide	Calcium thiosulphate	Free sulphur		
Dry lime-sulphur A	71.87	7.38	4.70	13.5 lbs.	9.70
Do	71.87	7.38	4.70	20 lbs.	14.37
Do	71.87	7.38	4.70	27 lbs.	19.40
Dry lime-sulphur B	64.80	8.81	11.15	12.5 lbs.	8.10
Do	64.80	8.81	11.15	19.5 lbs.	12.63
Do	64.80	8.81	11.15	25 lbs.	16.20
Dry lime-sulphur C	73.92	9.35	4.09	13.5 lbs.	9.97
Do	73.92	9.35	4.09	19 lbs.	14.04
Do	73.92	9.35	4.09	27 lbs.	19.95
Liquid lime-sulphur <sup>1</sup>	31.57	1.95	-----	6.66 gals.	22.75
Do	31.57	1.95	-----	3.25 gals.	11.10

<sup>1</sup> Sp. gr. 1.30; Baumé 33.4°.

On April 23, twigs were taken from all parts of the trees and a count of 1,000 hibernating scales was made for each plat. The results of these experiments are given in Table 3.

TABLE 3.—*Results of tests against the San Jose scale made on apple trees at Washington, Ind., in 1921*

Material used	Quantity in 50 gallons of spray	Hibernating scale	
		Dead	Control
Dry lime-sulphur A	Pounds	Per cent	Per cent
	13.5	75.2	14.5
	20	83.6	43.5
	27	80.4	32.4
Dry lime-sulphur B	12.5	78.3	25.2
	19.5	84.0	44.8
	25	83.1	41.7
Dry lime-sulphur C	13.5	71.2	.7
	19	89.6	64.1
	27	84.0	44.8
Liquid lime-sulphur	Gallons		
	6.66	88.4	60.0
Check	3.25	70.4	0
	( <sup>1</sup> )	71.0	-----

<sup>1</sup> Untreated.

The results shown in Table 3 indicate that at dilutions of 12.5 to 13.5 pounds to 50 gallons these dry lime-sulphurs were of no practical value against the San Jose scale, and when the strength was increased they did not furnish a practical control. Liquid lime-sulphur used at the rate of 3.25 gallons to 50 gallons of water, which is roughly equivalent to the weaker strength of the dry materials, was of no value.

#### EXPERIMENTS IN 1922

The experiments on apple trees in 1922 were conducted at Bicknell, Ind., in a scale-incrusted orchard, which was divided into plats of three to five record trees. The application was made on March 15, using a power sprayer which maintained a pressure of 250 to 275 pounds with one spray gun and one rod. From 9 to 10 gallons of

spray were used per tree. The composition and dilution of the essential materials in the sprays are shown in Table 4.

TABLE 4.—Composition of the dry calcium-sulphurs tested in experiments against the San Jose scale in 1922

Material used	Percentage of—			Quantity used in 50 gallons of spray	Quantity of polysulphides in 50 gallons
	Calcium poly-sulphides	Calcium thio-sulphate	Sulphur		
Dry lime-sulphur A.....	70.72	8.76	3.35	13.5	9.54
Do.....	70.72	8.76	3.35	20	14.14
Do.....	70.72	8.76	3.35	27	19.09
Dry lime-sulphur B.....	72.43	9.33	6.50	12.5	9.05
Do.....	72.43	9.33	6.50	19.8	14.34
Do.....	72.43	9.33	6.50	25	18.10
Dry lime-sulphur C.....	69.16	12.53	6.03	13.5	9.33
Do.....	69.16	12.53	6.03	20	13.83
Do.....	69.16	12.53	6.03	27	18.67
Liquid lime-sulphur <sup>1</sup> .....	31.71	1.80	-----	6.66	22.87
Do. <sup>1</sup> .....	31.71	1.80	-----	3.25	11.16
Liquid lime-sulphur <sup>2</sup> .....	30.59	1.88	-----	6.66	21.88
Do. <sup>2</sup> .....	30.59	1.88	-----	3.25	10.68

<sup>1</sup> Used in Mississippi; sp. gr. 1.30, Baumé 33.4°.

<sup>2</sup> Used in Indiana; sp. gr. 1.29, Baumé 32.6°.

On April 26 a careful count was made of 2,000 scales on twigs taken from all parts of the trees in each plat. On June 29, 80 linear inches of new wood from each plat were examined and the number of young scales of the first generation recorded. The results of these counts are given in Table 5.

TABLE 5.—Results of tests against the San Jose scale made on apple trees at Bicknell, Ind., in 1922

Material used	Quantity in 50 gallons of spray	Hibernating scales		Infestation of young scales on 80 linear inches new wood		
		Dead	Control	Total scale	Average per inch	Percentage of check
Dry lime-sulphur A.....	Pounds 13.5 20 27	Per cent 65.5 80.5 84.6	Per cent 23.3 56.6 65.7	3,801 2,882 2,459	47.51 36.02 30.73	42.8 32.5 27.7
Dry lime-sulphur B.....	12.5 19.8	69.8 75.1	32.8 44.6	5,008 3,684	62.60 46.05	56.4 41.5
Dry lime-sulphur C.....	13.5 20 27	60.7 78.5 81.3	12.6 52.2 58.4	2,471 4,806 2,279	30.88 60.07 28.49	27.8 54.2 25.7
Liquid lime-sulphur.....	Gallons 6.66 3.25	97.6 70.8	94.6 35.1	175 3,547	2.19 44.34	2.0 40.0
Check.....	Untreated.	55.0	-----	8,873	110.91	-----

#### APPLE TREES, 1922

In the 1922 experiments, as in those of 1921, the weaker solution failed to give any practical control of scale. Although a much higher percentage of control was obtained with the greater strengths, it was still much lower than that secured with liquid lime-sulphur and

can not be considered satisfactory. Liquid lime-sulphur at 3.25 gallons to 50 gallons of water was a little more effective than the weaker strengths of the dry lime-sulphurs.

These results are confirmed by the counts of young scales that settled on the new wood, since the average number of scales found on the twigs treated with the weaker solutions of the three dry lime-sulphurs was 25.9 times the number found in the liquid lime-sulphur plat and approximately 50 per cent of the number found on the untreated check. The strongest solutions of the dry materials gave an average of 59.7 per cent control and an average of 27 per cent as many young scales on the new wood as were found on the untreated checks.

#### PEACH TREES, 1922

The same spray materials (see Table 4) were tested against the San Jose scale on peach trees at Canton, Miss., where plats of five trees each were given a dormant spray on February 12 and 13. A barrel pump, maintaining a pressure of 100 to 125 pounds with a rod and disk nozzle, was used and an average of 1½ gallons of spray material was applied to each tree.

The count of hibernating scales was made on March 7 and the counts of the newly settled scales of the first and second generations were made from June 1 to 3 and from August 10 to 12. The results of these tests are given in Table 6.

TABLE 6.—*Results of tests against the San Jose scale on peach trees at Canton, Miss., in 1922*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scales on 80 linear inches new wood					
		Dead	Control	First generation			Second generation		
				Total scale	Average per inch	Per cent of check	Total scale	Average per inch	Per cent of check
Dry lime-sulphur A.....	Pounds	Per cent	Percent						
Dry lime-sulphur A.....	13.5	41.9	27.1	403	5.04	27.1	899	11.23	42.4
Dry lime-sulphur A.....	20	45.4	31.5	336	4.20	22.7	683	8.53	32.2
Dry lime-sulphur A.....	27	54.1	42.4	194	2.42	13.1	671	8.38	31.6
Dry lime-sulphur B.....	12.5	43.3	28.9	375	4.69	25.3	732	9.15	34.5
Dry lime-sulphur B.....	19.8	49.0	36.0	292	3.65	19.7	629	7.86	29.7
Dry lime-sulphur B.....	25	57.2	46.4	202	2.53	13.6	643	8.03	30.3
Dry lime-sulphur C.....	13.5	39.1	23.7	376	4.70	25.4	688	8.60	32.4
Dry lime-sulphur C.....	20	48.6	35.6	275	3.44	18.5	588	7.35	27.7
Dry lime-sulphur C.....	27	56.2	45.1	145	1.81	9.8	420	5.25	19.8
Liquid lime-sulphur.....	Gallons								
Liquid lime-sulphur.....	6.66	77.6	71.9	37	.46	2.5	117	1.46	5.5
Liquid lime-sulphur.....	3.25	31.2	13.8	437	5.46	29.5	600	7.50	28.3
Check.....	Untreated.	20.2	-----	1,483	18.54	-----	2,121	26.51	-----

In the experiments reported in Table 6 the weaker solutions of the dry lime-sulphur gave an average control of 26.6 per cent, and the stronger solutions 44.6 per cent, as compared with a control of 71.9 per cent for liquid lime-sulphur.

On the new wood the weaker strengths of the dry materials showed an average of 4.8 scales of the first generation per linear inch. The strongest solutions gave 2.25 per inch and the liquid lime-sulphur 0.46. The untreated checks showed an average of 18.5 scales per inch.

In the second generation the weaker strengths showed about 36.4 per cent and the greater strengths about 27 per cent, as many young scales as were found on the untreated check. These figures show an infestation 6.6 and 4.9 times as great as where the liquid lime-sulphur was used.

## EXPERIMENTS IN 1923

## APPLE TREES

The tests on apple trees were conducted at Stuart, Va., in an 18-year-old orchard moderately infested with scale. Plots of five trees each were given a dormant spray on March 8, using a power sprayer which maintained a pressure of 225 pounds with one spray gun. An average of 12 gallons per tree was used. The composition and dilutions of the essential materials in these sprays are shown in Table 7.

TABLE 7.—Composition of the dry calcium-sulphurs tested in experiments against the San Jose scale in 1923

Material used	Percentage of—			Quantity used in 50 gallons of spray	Pounds of poly-sulphide in 50 gallons of spray
	Calcium poly-sulphides	Calcium thio-sulphate	Free sulphur		
Dry lime-sulphur A	72.09	9.45	1.42	15	10.81
Do	72.09	9.45	1.42	27	19.46
Dry lime-sulphur B	68.68	11.18	6.40	15	10.30
Do	68.68	11.18	6.40	27	18.54
Dry lime-sulphur C	78.78	9.00	.86	15	11.81
Do	78.78	9.00	.86	26	20.48
Liquid lime-sulphur <sup>1</sup>	30.34	2.04	-----	Gallons 6.66	21.53

<sup>1</sup> Specific gravity 1.28, Baumé 31.8°.

The count of hibernating scales was made on April 11 and the first generation of the scale on new wood was counted on June 27. The results of these experiments are shown in Table 8.

TABLE 8.—Results of tests against the San Jose scale made on apple trees at Stuart, Va., in 1923

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches new wood		
		Dead	Control	Total scale	Average per inch	Percent-age of check
Dry lime-sulphur A	Pounds 15	Per cent 48.7	Per cent 33.6	289	3.61	35.9
	27	63.1	60.0	121	1.55	15.4
Dry lime-sulphur B	15	59.2	47.2	294	3.68	36.6
	27	74.6	67.1	117	1.46	14.6
Dry lime-sulphur C	15	50.0	35.3	258	3.23	32.1
	26	71.4	63.0	132	1.65	16.4
Liquid lime-sulphur	Gallons 6.66	97.0	96.1	51	.64	6.34
Check	(1)	22.7	-----	804	10.05	-----

<sup>1</sup> Untreated.

In the experiments reported in Table 8 the dry lime-sulphurs at the rate of 15 pounds to 50 gallons of water gave an average control of 38.7 per cent, and at 26 and 27 pounds to 50 gallons an average control of 63.4 per cent as compared with 96.1 per cent for liquid lime-sulphur.

The average number of young scales per linear inch of new wood for the same experiments was 3.5, 1.55, and 0.64. Although the number of young scales found per inch of new wood was rather small, it should be noted that the 15-pound treatment allowed 5.5 times as many scales to settle as did the liquid lime-sulphur; and the 26 and 27 pound treatments 2.4 times as many.

#### PEACH TREES, 1923

The same materials (see Table 7) were tested against the San Jose scale on peach trees at Canton, Miss., in the same orchard that was used in 1922, although the infestation was not so heavy as that of the previous season. From five to seven trees were used per plat, and these were sprayed on February 14, using a barrel pump which maintained a pressure of 100 to 125 pounds with a rod and disk nozzle. An average of 1½ gallons per tree was applied.

The hibernating scales were counted on March 23 and the counts of the first and second generations were made on June 27 and October 4. The results of these experiments are shown in Table 9.

TABLE 9.—*Results of tests against the San Jose scale on peach at Canton, Miss., in 1923*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches new wood						
				First generation			Second generation			
		Dead	Con- trol	Total scales	Aver- age per inch	Per- centage of check	Total scales	Aver- age per inch	Per- centage of check	
Dry lime-sulphur A-----	Pounds	15	53.2	44.2	215	2.69	21.0	627	7.84	49.7
		27	69.7	63.8	157	1.96	15.4	425	5.31	33.7
Dry lime-sulphur B-----		15	55.9	47.4	206	2.58	20.2	487	6.09	38.6
		27	75.0	70.2	151	1.89	14.8	249	3.11	19.7
Dry lime-sulphur C-----		15	50.7	41.2	242	3.03	23.7	500	6.25	39.6
	Gallons	26	70.1	64.3	136	1.70	13.3	289	3.61	22.9
Liquid lime-sulphur-----		6.66	96.4	95.7	39	.49	3.8	92	1.15	7.3
Check-----	(1)		16.2	-----	1,022	12.78	-----	1,262	15.78	-----

<sup>1</sup> Untreated.

When used on peach trees at the rate of 15 pounds to 50 gallons of water the average control obtained with the dry materials was 44.27 per cent. When the strength was increased to 26 or 27 pounds the control rose to 66.10 per cent, but this does not compare very favorably with the 95.7 per cent control obtained with liquid lime-sulphur concentrate. The 15-pound applications showed an average of 42.6 per cent and the 27-pound spray an average of 25.4 per cent as many young scales of the second generation as were found on the untreated check, which is 5.9 and 3.5 times as many as were present in the liquid lime-sulphur plat.

## DISCUSSION OF FIELD EXPERIMENTS WITH DRY CALCIUM-SULPHURS

The experiments already described show that when these dry lime-sulphurs were used at dilutions from 12.5 to 15 pounds to 50 gallons of water the average control on hibernating scale was only 29.16 per cent. When the strength was increased to 25 or 27 pounds the average control was increased to 54.68 per cent. In the same series of tests standard liquid lime-sulphur gave an average control of 83.66 per cent. These figures show that the dry materials tested did not give a control against hibernating scale which approximated that obtained with liquid lime-sulphur, or one that could be considered of very much practical value.

If the efficiency of these dry substitutes is measured by the number of young scales that settle on the new wood, the same lack of control is found. On apple trees the weaker sprays showed an average of 2,409 scales of the first generation on each 80 linear inches of new wood examined, or 30 scales to the inch. The stronger sprays gave an average of 1,263.6 scales per 80 inches, or 15.8 per inch. These treatments should be compared with liquid lime-sulphur which allowed an average of 1.4 scales per inch to settle.

The counts of second-generation scales on peach showed an average of 8.19 scales per inch of new wood where the weaker sprays were used, 5.61 for the stronger sprays, and 1.3 for liquid lime-sulphur.

These experiments cover a variety of conditions, since they were carried on in Indiana, Virginia, and Mississippi. In 1922 the orchards treated were incrusted and the scale was increasing very rapidly, whereas in 1923 orchards only moderately infested, in which the scale was not increasing very rapidly, were used. Although the dry lime-sulphurs, in all cases, show some effect on the scale, the apparent control was never great enough to justify their being used as remedies for the San Jose scale under general orchard conditions.

S. A. Forbes has estimated (11, p. 549) that the theoretical number of the progeny from one hibernating female San Jose scale might, under ideal or optimum conditions, in one season, reach the astonishing total of 32,791,472. If the actual increase under natural conditions is even as low as 1 per cent of the theoretical it is still very evident that a treatment which does not kill more than 50 per cent or 60 per cent of the hibernating scales, and allows from three to six scales to settle on each inch of the new wood, can not be considered satisfactory, since it would not prevent an immediate reinfestation and the resulting damage to the trees.

## EXPERIMENTS IN 1924

The experiments conducted in 1924 were primarily for the purpose of determining the relative value of the ingredients found by chemical analysis to be present in the dry and liquid lime-sulphurs.

Based on the analyses of more than 100 commercial lime-sulphur solutions the average molecular ratio  $\frac{\text{Polysulphide sulphur}}{\text{Polysulphide calcium}}$  is 4.68, indicating a predominating percentage of the higher sulphide,  $\text{CaS}_5$ , whereas from the analyses of 38 samples of dry lime-sulphur the molecular ratio  $\frac{\text{Polysulphide sulphur}}{\text{Polysulphide calcium}}$  is 3.53, indicating that the polysulphides are mainly the lower sulphides,  $\text{CaS}_4$  and  $\text{CaS}_3$ .

From these observations it was thought that there might be a difference in the efficacy of the products on account of the fact that the polysulphide sulphur in the dry lime-sulphur was not in as high a ratio to the polysulphide calcium as the polysulphide sulphur to polysulphide calcium in the lime-sulphur solution.

With this in mind, solutions were prepared having the same molecular ratio of sulphur and lime as calcium trisulphide, calcium tetrasulphide, and calcium pentasulphide. These solutions were diluted so that the total polysulphide content, when applied as a spray, was the same as the total polysulphide content of the lime-sulphur solution and dry lime-sulphur when applied as sprays. Calcium thiosulphate, calcium sulphite, and sulphur were also prepared and tested.

The essential materials used and the results of these experiments are shown in Table 10.

TABLE 10.—*Effectiveness of the ingredients of dry and liquid lime-sulphur used in experiments against the San Jose scale*

Experiment No.	Material used	Dilution	Dead	Control
1	Liquid lime-sulphur (32 per cent calcium polysulphide).	6.66 gallons to 50 gallons.....	Per cent 95.95	Per cent 94.98
2	Same, with 1.11 pounds of sugar to 50 gallons.....	do.....	95.90	94.92
3	Same, with 10 pounds of sugar to 50 gallons.....	do.....	93.00	91.33
4	Liquid lime-sulphur A.....	Equivalent to No. 1 <sup>1</sup> .....	89.75	87.30
5	Same, with 1.11 pounds of sugar to 50 gallons.....	do.....	88.00	85.14
6	Dry lime-sulphur A.....	do.....	50.40	38.58
7	do.....	do.....	59.0	35.94
8	Same with all sludge removed.....	do.....	96.0	93.75
9	Calcium pentasulphide ( $\text{CaS}_5$ ) <sup>2</sup> .....	do.....	93.10	91.46
10	do.....	do.....	95.20	94.27
11	Calcium tetrasulphide ( $\text{CaS}_4$ ) <sup>3</sup> .....	do.....	89.5	87.00
12	do.....	do.....	93.5	92.24
13	Calcium trisulphide ( $\text{CaS}_3$ ) <sup>3</sup> .....	do.....	43.4	29.91
14	Calcium thiosulphate.....	1 ounce to 1 gallon.....	13.4	0
15	do.....	24 ounces to 1 gallon.....	18.45	0
16	Calcium sulphite.....	½ ounce to 1 gallon.....	19.37	.15
17	do.....	12 ounces to 1 gallon.....	16.55	0
18	Sulphur.....	Equivalent to No. 1 <sup>1</sup> .....	19.45	.25
19	Check, untreated.....	.....	19.25	.....

<sup>1</sup> Based on polysulphide-sulphur content.

<sup>2</sup> Check, 36 per cent dead.

<sup>3</sup> Calcium polysulphides are so prepared that the molecular ratio of calcium to sulphur equals 1 to 5, 1 to 4, and 1 to 3, respectively.

\* 1923 experiments; check, 16.2 per cent dead.

With the exception of Nos. 7, 8, 10, and 12, the experiments considered in Table 10 were carried on in a moderately infested peach orchard in Opelika, Ala. A dormant application was made on January 28 and 29, using a wheelbarrow sprayer having a vertical agitator and maintaining a pressure of about 100 pounds. The counts of hibernating scales were made on March 7, 8, 9, and 10.

Experiments 7 and 8 were made on small, badly infested peach trees near Vienna, Va. The sprays were applied with a knapsack sprayer on March 17 and the scale count was made on April 17.

Experiments 10 and 12 were a part of the 1923 series on apple trees, the details of which are given on pages 11 and 12.

Experiments 2, 3, and 5 mentioned in Table 10, which were made with liquid lime-sulphur and granulated sugar, indicate that the

addition of sugar, at the rate of 1.11 pounds and 10 pounds to 50 gallons of spray solution, does not materially reduce the effectiveness against the San Jose scale.

Experiments 6 and 7 show that dry lime-sulphur A, even when used at a strength (33 pounds to 50 gallons) which furnishes an amount of polysulphide sulphur equivalent to that found in standard liquid lime-sulphur, can not be considered an effective remedy against this scale.

Dry lime-sulphur A, at the rate of 33 pounds to 50 gallons of water, was used for experiments 7 and 8.

After this material had been mixed a part of the solution was carefully filtered to remove the insoluble portions. One-half of this filtrate was used for experiment 8. Half of the insoluble sludge that had been removed was added to the remaining filtrate and this was used in experiment 7.

The 93.75 per cent control obtained with the filtered material (experiment 8) compared with the 35.94 per cent control obtained with the part containing the sludge (experiment 7) indicates that the insoluble matter present, when this dry lime-sulphur was used at the rate of 33 pounds to 50 gallons of water, greatly reduced the effectiveness of the spray.

This detrimental effect of the sludge also explains why, in the field experiments when the amount of dry lime-sulphur was doubled, there was not a corresponding increase in effectiveness.

The experiments with the three polysulphides, prepared to represent calcium pentasulphide, tetrasulphide, and trisulphide, respectively, show that the pentasulphide and the tetrasulphide furnish a satisfactory control of the scale and the trisulphide is of very little practical value. The calcium thiosulphate, calcium sulphite, and free sulphur, at the strength used, were found to be of no value.

#### DISCUSSION OF RESULTS

The foregoing experiments show that the three dry lime-sulphurs tested were not satisfactory remedies against the San Jose scale, even when used in excessive quantities.

A study of the chemical analyses of the dry and liquid lime-sulphurs shows the following differences:

The dry lime-sulphurs contain sulphur and calcium in the proportions to form calcium trisulphide ( $\text{CaS}_3$ ) and calcium tetrasulphide ( $\text{CaS}_4$ ), they contain considerable amounts of calcium thiosulphate and free sulphur, and, at increased strengths, an objectionable quantity of insoluble sludge.

Liquid lime-sulphur contains polysulphide sulphur in such proportions as to form calcium tetrasulphide ( $\text{CaS}_4$ ) and calcium pentasulphide ( $\text{CaS}_5$ ), with the latter predominating; very little calcium thiosulphate, no free sulphur, and no insoluble sludge.

If Shafer's theory (23) that the efficacy of a lime-sulphur solution against scale insects is due to an oxidizing action is correct, it naturally follows that the higher polysulphides ( $\text{CaS}_5$  and  $\text{CaS}_4$ ) would be more effective than the lower ( $\text{CaS}_3$ ) since, per molecule, they would furnish a larger quantity of nascent sulphur to act as an oxidizing agent.

The experiments given in Table 10 show that the calcium thiosulphate and the free sulphur are of no practical value against scale.

Experiments 6 and 7 in Table 10 show that the presence of a considerable amount of insoluble sludge greatly reduces the effectiveness of these dry preparations. It is thus clearly evident that there are adequate chemical and physical reasons for the failure of these dry lime-sulphurs to control the San Jose scale.

#### SUMMARY

(1) The dry lime-sulphurs used were found to be of little practical value against the San Jose scale.

(2) The addition of sugar did not materially reduce the effectiveness of liquid lime-sulphur.

(3) Calcium polysulphides corresponding to the pentasulphide and tetrasulphide were found to be effective and that corresponding to the trisulphide of very little value against the San Jose scale.

(4) Calcium thiosulphate, calcium sulphite, and free sulphur were of no practical value.

(5) The lack of effectiveness in the dry lime-sulphurs may have been caused by the following:

(a) When the water is removed from liquid lime-sulphur to produce the dry calcium-sulphur, the calcium polysulphides are changed from the mixture of polysulphides 5 ( $\text{CaS}_5$ ) and 4 ( $\text{CaS}_4$ ) with the 5 predominating, which is found in liquid lime-sulphur, to a mixture of polysulphides 4 ( $\text{CaS}_4$ ) and 3 ( $\text{CaS}_3$ ) in approximately equal proportions. This change would, according to the experiments given in Table 10, reduce the effectiveness of the dry calcium sulphurs since the higher polysulphides were found to be more effective than the lower ones.

(b) The calcium thiosulphate, sulphite, and free sulphur present are not effective.

(c) The presence of a considerable amount of insoluble sludge apparently reduces the effectiveness of these materials against the San Jose scale.

#### DRY SODIUM-SULPHUR COMPOUNDS

The dry sodium-sulphur compounds, which are also sold as substitutes for lime-sulphur solution, are likewise of comparatively recent development. Patents<sup>11</sup> covering several methods for the manufacture of these preparations have been obtained. However, the method most generally employed consists in heating or fusing together sulphur and sodium carbonate (soda ash), or sulphur and caustic soda.

#### CHEMICAL COMPOSITION

The results of the chemical analysis of 15 samples of sodium-sulphur compounds, obtained in the open market and including the principal brands, are given in Table 11.

<sup>11</sup> United States Nos. 1,044,452; 1,132,476; and 1,457,652.

TABLE 11.—*The chemical composition of representative dry sodium-sulphurs*

Sample No.	Sodium polysulphide <sup>1</sup>	Sodium thiosulphate <sup>2</sup>	Free sulphur	Other ingredients by difference <sup>3</sup>	Sample No.	Sodium polysulphide <sup>1</sup>	Sodium thiosulphate <sup>2</sup>	Free sulphur	Other ingredients by difference <sup>3</sup>
	Per cent	Per cent	Per cent	Per cent		Per cent	Per cent	Per cent	Per cent
1-----	55.11	41.93	0.13	2.83	9-----	41.13	50.52	0.67	7.68
2-----	58.21	39.20	.54	2.05	10-----	40.91	49.43	.35	9.31
3-----	72.00	7.67	.99	19.34	11-----	43.55	29.39	20.03	7.03
4-----	60.18	25.87	1.25	12.70	12-----	41.99	41.45	10.33	6.23
5-----	52.83	40.74	.48	5.95	13-----	61.53	29.35	.34	8.78
6-----	56.84	36.56	3.38	3.22	14-----	2.12	60.00	18.94	18.94
7-----	68.30	10.80	1.39	19.51	15-----	10.96	61.60	17.54	9.90
8-----	55.13	40.80	1.56	2.51					

<sup>1</sup> Polysulphide sulphur calculated to sodium polysulphide ( $\text{Na}_2\text{S}_x$ ).<sup>2</sup> Thiosulphate sulphur calculated to sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3$ ).<sup>3</sup> Mainly water.

It will be noted that these products are extremely variable in composition, the sodium polysulphide varying from 2.12 to 72 per cent, sodium thiosulphate from 7.67 to 61.6 per cent and free sulphur from 0.13 to 20 per cent.

A comparison of the composition of the liquid lime-sulphurs and the sodium-sulphur compounds shows that the latter contain roughly about  $1\frac{1}{2}$  times as much polysulphides; from 15 to 25 times as much thiosulphates; and, in addition, varying quantities of free sulphur, which does not occur in liquid lime-sulphur solution.

The analysis of more than 100 commercial lime-sulphur solutions indicates the presence of a predominating percentage of the pentasulphide ( $\text{CaS}_5$ ). In the case of the sodium-sulphur preparations they show such wide variations in composition that no general statement can be made in regard to the sodium polysulphide that may predominate; although some of them apparently contain sulphur in the pentasulphide form.

Table 12 gives the chemical composition of the dry sodium-sulphur preparations that were tested in experiments against the San Jose scale. These experiments were carried on under the same general conditions as those described for the dry calcium-sulphur tests which have already been discussed. For the details of these tests reference should be made to the dry calcium-sulphur experiments under the corresponding locality and date.

TABLE 12.—*The chemical composition of the dry sodium sulphur preparations tested*

Sample No.	Percentage of—					Pounds used in 50 gallons of spray	Pounds of poly-sulphides in 50 gallons
	Sodium poly-sulphides	Sodium thio-sulphate	Free sulphur	Sodium sulphate	Insoluble residue and water		
1-----	59.71	25.79	2.54	3.72	8.24	12.5	7.46
1-----	59.71	25.79	2.54	3.72	8.24	19.	11.34
1-----	59.71	25.79	2.54	3.72	8.24	25	14.92
2-----	61.26	23.95	6.54	6.09	2.16	12.5	7.65
2-----	61.26	23.95	6.54	6.09	2.16	19	11.63
2-----	61.26	23.95	6.54	6.09	2.16	25	15.31
3-----	60.53	22.76	3.07	9.21	4.43	12.5	7.56
3-----	60.53	22.76	3.07	9.21	4.43	27	16.34

EXPERIMENTS IN 1921<sup>12</sup>

In these tests dry sodium-sulphur No. 1 was used against the San Jose scale on apple trees at Washington, Ind. The results of these tests are shown in Table 13.

TABLE 13.—*Results of tests with dry sodium-sulphur sprays against the San Jose scale on apple trees at Washington, Ind., in 1921*

Material used	Amount in 50 gallons of spray	Hibernating scale	
		Dead	Control
Sodium-sulphur No. 1.....	Pounds	Per cent	Per cent
Do.....	12.5	79.0	27.6
Do.....	19	83.8	44.1
Do.....	25	84.4	46.2
Liquid lime-sulphur <sup>1</sup> .....	Gallons		
Check.....	6.66	88.4	60.0
		71.0	-----

<sup>1</sup> Baumé 33.4°.

The tests recorded in Table 13 show clearly that, even when a large percentage of the scales die from natural causes, dry sodium sulphur does not serve as an effective remedy. They also show that when the conditions are such that the standard liquid lime-sulphur treatment falls below what can be considered an effective control this preparation is even less effective. Although increasing the dosage increased the efficiency of the dry sodium-sulphur, in no case was an effective control obtained.

EXPERIMENTS IN 1922<sup>13</sup>

Dry sodium-sulphur No. 2 was tested against the San Jose scale on apple at Bicknell, Ind., and on peach at Canton, Miss. The results of these tests are given in Tables 14 and 15.

TABLE 14.—*Results of tests with dry sodium-sulphur sprays against the San Jose scale on apple trees at Bicknell, Ind., in 1922*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood		
		Dead	Control	Number of scales	Average per inch	Percent- age of check
Sodium-sulphur No. 2.....	Pounds	Per cent	Per cent			
Do.....	12.5	63.4	18.7	4,086	51.1	46.0
Do.....	19	73.7	41.6	2,523	31.6	28.5
Do.....	25	85.4	67.6	1,140	14.3	12.8
Liquid lime-sulphur <sup>1</sup> .....	Gallons					
Check.....	6.66	97.6	94.7	175	2.19	2.0
		55	-----	8,873	110.91	-----

<sup>1</sup> Baumé 32.6°.

Table 14 gives the results of tests in an orchard where the scale was increasing very rapidly. Under these adverse conditions liquid lime-sulphur gave a very good control but the dry sodium-sulphur spray

<sup>12</sup> For details of these tests see p. 7-8.

<sup>13</sup> For details of these tests see pages 8-11.

even at the rate of 25 pounds to 50 gallons could not be considered satisfactory. The weakest spray not only failed to give a good control of hibernating scale, but allowed almost half as many young scales to settle on the new wood as were counted on the untreated trees.

TABLE 15.—*Results of tests with dry sodium-sulphur sprays against the San Jose scale on peach trees at Canton, Miss., in 1922*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood					
				First generation			Second generation		
		Dead	Control	Number of scales	Average per inch	Percent- age of check	Number of scales	Average per inch	Percent- age of check
Sodium-sulphur No. 2.....	Pounds 12.5 19 25	Per cent 34.4 48.9 58.8	Per cent 17.8 36.0 48.4	302 278 178	4.53 3.48 2.23	24.5 18.7 12.0	783 540 419	9.70 6.75 5.24	36.9 25.5 19.8
Liquid lime-sulphur 1.....	Gals. 6.66	77.6 20.2	71.9	37 1,483	.46 18.5	2.5	117 2,121	1.46 26.51	5.5
Check.....									

<sup>1</sup> Baumé 33.4°.

In the experiments reported in Table 15 the dry sodium-sulphur failed to show a control that could be considered satisfactory, even when used at the rate of 25 pounds to 50 gallons of water. Although the liquid lime-sulphur was not satisfactorily effective, it gave much better control than did the dry substitute.

#### EXPERIMENTS IN 1923 <sup>14</sup>

The tests with dry sodium-sulphur No. 3 were made at Stuart, Va., and Canton, Miss. The results of these experiments are given in Tables 16 and 17.

TABLE 16.—*Results of tests with dry sodium-sulphur sprays against the San Jose scale on apple trees at Stuart, Va., in 1923*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood		
		Dead	Control	Number of scales	Average per inch	Percent- age of check
Sodium-sulphur No. 3.....	Pounds 12.5 27	Per cent 55.6 72.8	Per cent 42.6 64.8	216 145	2.7 1.81	26.9 18.0
Liquid lime-sulphur 1.....	Gallons 6.66	97.0 22.7	96.1	39 804	.49 10.05	4.9
Check.....						

<sup>1</sup> Baumé 31.8°.

The experiments reported in Table 16 indicate the same lack of control that has been noted in the earlier tests. Although the

<sup>14</sup> For details of these experiments see p. 11-12.

greater strength was more effective this increase was not in the same ratio as the increase in dosage.

The infestation in this orchard was not very heavy but even under these favorable conditions the 12.5-pound dosage allowed over 5.5 times as many young scales to settle on the new wood as did the liquid lime-sulphur.

TABLE 17.—*Results of tests with dry sodium-sulphur sprays against the San Jose scale on peach trees at Canton, Miss., in 1923*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood					
				First generation			Second generation		
		Dead	Control	Number of scales	Aver- age per inch	Per- centage of check	Number of scales	Aver- age per inch	Per- centage of check
Sodium-sulphur No. 3.....	Pounds 12.5 26.625	Percent 53.0 71.0	Percent 43.9 65.4	353 247	4.41 3.09	34.5 24.2	525 365	6.56 4.56	41.6 28.9
Liquid lime-sulphur <sup>1</sup> .....	Gallons 6.66	96.4 16.2	95.7	39 1,022	.49 12.78	3.8	92 1,262	1.15 15.78	7.3
Check.....									

<sup>1</sup> Baumé 31.8°.

The experiments discussed in Table 17 were carried on during a season when the scale was not increasing so rapidly as in 1922, and all of the treatments gave better control than in the previous season, but the relative efficiency is much the same.

It is of interest to note that in the first experiment, in spite of the 43.9 per cent control, the second count of young scales showed 41.6 per cent, as many as were found on the untreated checks.

#### DISCUSSION OF RESULTS

When used at the strength ordinarily employed (12.5 pounds to 50 gallons) the average control on hibernating scales was 30 per cent. When this dosage was increased to 25 to 27 pounds the control only rose to 58.3 per cent. In the same set of experiments liquid lime-sulphur gave an average control of 83.6 per cent.

These figures are supported by the counts of first and second generations of young scales on the new wood, since in every case many more were found in the sodium-sulphur plats than where liquid lime-sulphur was used.

#### SUMMARY

The experiments with the dry sodium-sulphur sprays show, on the whole, results that are very similar to those obtained with the dry calcium-sulphurs. In no case were they as effective as liquid lime-sulphur, even when used at greatly increased strengths, and it can not be considered that they furnished a satisfactory control of the San Jose scale.

These experiments covered a period of three years, and were carried on in three different States under widely varying conditions.

## DRY BARIUM-SULPHUR COMPOUNDS

The dry barium-sulphur compounds, which are sold as substitutes for lime-sulphur solution, are of comparatively recent origin. Several methods for their preparation have been developed, and patents<sup>15</sup> granted covering some of these processes, but all, or practically all, of those produced at the present time consist simply of a mixture of "black ash" (a crude barium-sulphide, BaS, made by heating barium sulphate with coal in a furnace) and sulphur.

## CHEMICAL COMPOSITION

Commercial dry barium-sulphur contains—in addition to barium sulphide and sulphur—chemically combined water, some barium sulphate, siliceous material, carbon, and small quantities of other impurities. These impurities are objectionable in that they decrease the percentage of active ingredients, impede their action, tend to clog the nozzle of the spraying apparatus, and the graphitelike residue from the coal is very destructive, on account of its abrasive action, to the spray pumps and nozzles.

The results of analyses of nine samples of commercial dry barium-sulphurs purchased in various parts of the United States are given in Table 18.

TABLE 18.—*Chemical composition of representative dry barium-sulphurs*

Sample No.	Barium-sulphide (BaS)	Free sulphur	Barium-thiosulphate (BaS <sub>2</sub> O <sub>3</sub> )	Other ingredients, by difference <sup>1</sup>	Sample No.	Barium-sulphide (BaS)	Free sulphur	Barium-thiosulphate (BaS <sub>2</sub> O <sub>3</sub> )	Other ingredients, by difference <sup>1</sup>
	Per cent	Per cent	Per cent	Per cent		Per cent	Per cent	Per cent	Per cent
1-----	34.81	38.64	3.46	23.09	6-----	38.00	34.01	3.97	24.02
2-----	32.84	36.24	4.32	26.60	7-----	39.76	34.88	2.96	22.40
3-----	35.30	32.03	4.55	28.12	8-----	39.41	33.66	1.28	25.65
4-----	46.66	37.11	1.98	14.25	9-----	38.10	34.60	4.09	23.21
5-----	41.22	31.05	7.47	20.26	Gen. av.---	38.46	34.69	3.79	23.07

<sup>1</sup> Chiefly barium sulphate, siliceous material, carbon, and moisture.

The dry barium-sulphurs, when diluted according to directions (12 to 14 pounds to 50 gallons of water), leave an insoluble residue which on filtering and drying at 105° C. amounts to about 34 per cent of the product. The soluble portion has a specific gravity of about 1.014 and contains approximately 2 per cent of barium-polysulphide in solution. The ratio of sulphur to barium indicates that the polysulphide is largely the tetrasulphide (BaS<sub>4</sub>). This will furnish approximately 8.5 pounds of barium-polysulphide in each 50 gallons of spray material.

Commercial liquid lime-sulphur (32° B.) at a dilution of 6.66 gallons to 50 gallons of water (1 to 7.5), which was the strength used in the tests here reported, will furnish approximately 22.8 pounds of calcium-polysulphides in each 50 gallons of spray material.

The experiments with dry barium-sulphur were a part of the series of tests with the various dry substitutes for liquid lime-sulphur. These tests were made under the same general conditions as those already described.

<sup>15</sup> United States Nos. 1,263,856 and 1,457,652.

Table 19 gives the chemical composition of the essential materials in the dry barium-sulphur sprays used against the San Jose scale.

TABLE 19.—*The chemical composition of the dry barium-sulphur preparations tested*

Sample No.	Percentage of—				Pounds used in 50 gallons of spray	Pounds of sulphides in 50 gallons
	Barium-sulphide	Barium-thiosulphate	Free sulphur	Barium-sulphate and siliceous material		
1.....	41.22	7.47	31.05	<sup>1</sup> 18.32	13	5.35
1.....	41.22	7.47	31.05	<sup>1</sup> 18.32	19.5	8.03
1.....	41.22	7.47	31.05	<sup>1</sup> 18.32	26	10.71
2.....	38.00	3.97	34.01	24.43	13	4.94
2.....	38.00	3.97	34.01	24.43	19.5	7.41
2.....	38.00	3.97	34.01	24.43	26	9.88
3.....	39.41	1.28	33.66	25.14	14	5.51
3.....	39.41	1.28	33.66	25.14	31	12.21

<sup>1</sup> 1.94 per cent undetermined.

#### EXPERIMENTS IN 1921<sup>16</sup>

Dry barium-sulphur No. 1 was tested against the San Jose scale on apple trees at Washington, Ind. The results of these tests are shown in Table 20.

TABLE 20.—*Results of tests with dry barium-sulphur sprays against the San Jose scale on apple trees at Washington, Ind., in 1921*

Material used	Quantity in 50 gallons of spray	Hibernating scale	
		Dead	Control
Barium-sulphur No. 1.....	Pounds	Per cent	Per cent
Do.....	13	75.2	14.5
Do.....	19.5	80.0	31.0
	26	91.6	71.0
Liquid lime-sulphur <sup>1</sup> .....	Gallons		
Check.....	6.66	88.4	60.0
	71.0		

<sup>1</sup> Baumé 33.4°.

The above experiments show that at the rate of 13 or 19.5 pounds to 50 gallons of water this material did not furnish a satisfactory control of the scale. When 26 pounds was used, it apparently gave much better results than the liquid lime-sulphur, but, since such effectiveness was not shown in any of the later experiments, it seems probable that some other factor influenced these results.

#### EXPERIMENTS IN 1922<sup>17</sup>

Dry barium-sulphur No. 2 was tested against the San Jose scale on apple at Bicknell, Ind., and on peach at Canton, Miss. The results of these tests are shown in Tables 21 and 22.

<sup>16</sup> For general conditions in these experiments see pp. 7-8.

<sup>17</sup> For details of these experiments see pp. 8-11.

TABLE 21.—*Results of tests with dry barium-sulphur sprays against the San Jose scale on apple trees at Bicknell, Ind., in 1922*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood (first generation)		
		Dead	Control	Number of scales	Average per inch	Percent- age of check
Barium-sulphur No. 2.....	Pounds	Per cent	Per cent			
13.....	13	72.1	38.0	3,796	47.45	42.8
".....	19.5	81.9	59.8	2,296	28.70	25.9
Do.....	26	86.9	70.8	1,181	14.76	13.3
Liquid lime-sulphur <sup>1</sup> .....	Gallons					
Check.....	6.66	97.6	94.7	175	2.19	2.0
		55.0	-----	8,873	110.91	-----

<sup>1</sup> Baumé 32.6°.

The results given in Table 21 show that dry barium-sulphur, even when used at the rate of 26 pounds to 50 gallons of water, did not furnish a control of hibernating scale that could be considered effective, or one that approached the effectiveness of the liquid lime-sulphur. If the value of the treatment is measured by the number of young scales that settle on the new wood the same lack of control is shown since 13 pounds to 50 gallons allowed almost half as many young scales per inch as settled on the untreated check and over 21 times as many as were found in the liquid lime-sulphur plat.

Even the maximum strength showed over six times as many young scales as were present on the twigs sprayed with standard liquid lime-sulphur.

TABLE 22.—*Results of tests with dry barium-sulphur sprays against the San Jose scale on peach trees at Canton, Miss., in 1922*

Material used	Quan- tity in 50 gal- lons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood					
		First generation			Second generation				
		Dead	Con- trol	Num- ber of scales	Aver- age per inch	Per- centage of check	Num- ber of scales	Aver- age per inch	Per- centage of check
Barium-sulphur No. 2.....	Lbs. 13.5	Per cent 42.4	Per cent 27.8	343	4.29	23.1	573	7.16	27.0
Liquid lime-sulphur <sup>1</sup> .....	Gals. 6.66	77.6	71.9	37	.46	2.42	117	1.46	5.5
Check.....		20.2	-----	1,483	18.50	-----	2,121	26.51	-----

<sup>1</sup> Baumé 32.6°.

Only one strength of the dry barium-sulphur spray was used in these experiments, and this failed to show an effective control when either the hibernating scale or the young scale on new wood is considered. Although the liquid lime-sulphur gave rather poor control, it was relatively much better than the dry material.

EXPERIMENTS IN 1923<sup>18</sup>

The tests with dry barium-sulphur No. 3 were made at Stuart, Va., and Canton, Miss. The results of these experiments are shown in Tables 23 and 24.

TABLE 23.—*Results of tests with dry barium-sulphur sprays against the San Jose scale on apple trees at Stuart, Va., in 1923*

Material used	Quantity in 50 gallons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood (first generation)		
		Dead	Control	Number of scales	Average per inch	Percent- age of check
		Pounds	Per cent			
Barium sulphur No. 3.....	14	54.9	41.7	233	2.91	28.9
Do.....	31	79.6	73.6	148	1.85	18.4
Liquid lime-sulphur <sup>1</sup> .....	Gallons 6.66	97.0	96.1	51	.64	6.3
Check.....		22.7	-----	804	10.05	-----

<sup>1</sup> Baumé 31.8°.

The tests reported in Table 23, although made where the scale was not very abundant, show the same lack of control, in spite of the fact that, in one case, the barium-sulphur was used at the rate of 31 pounds to 50 gallons. Even this strength showed 22.5 per cent less control on the hibernating scale and almost three times as many young scales per inch when compared with the trees sprayed with liquid lime-sulphur.

TABLE 24.—*Results of tests with dry barium-sulphur sprays against the San Jose scale on peach trees at Canton, Miss., in 1923*

Material used	Quantity in 50 gal- lons of spray	Hibernating scale		Infestation of young scale on 80 linear inches of new wood					
				First generation			Second generation		
		Dead	Con- trol	Num- ber of scales	Aver- age per inch	Per- cent- age of check	Num- ber of scales	Aver- age per inch	Per- cent- age of check
Barium-sulphur No. 3.....	Pounds 14	Per cent 56.7	Per cent 48.3	298	3.73	29.2	468	5.85	37.1
Do.....	31	71.4	65.9	173	2.16	17	231	2.89	18.3
Liquid lime-sulphur <sup>1</sup> .....	Gallons 6.66	96.4	95.7	39	.49	3.8	92	1.15	7.3
Check.....		16.2	-----	1,022	12.78	-----	1,262	15.78	-----

<sup>1</sup> Baumé 31.8°.

The experiments in Canton, Miss., show the same lack of control by barium-sulphur that was noted in previous tests. A strength of 31 pounds to 50 gallons gave a control of only 65.9 per cent on the hibernating scales and allowed 4.5 times as many scales to settle on the new wood as did the standard liquid lime-sulphur.

<sup>18</sup> Details of these experiments are given on pages 11 and 12.

## DISCUSSION OF RESULTS

When used at the rate of 13 or 14 pounds to 50 gallons the dry barium-sulphur gave an average of only 34 per cent control of hibernating scale. When the strength was increased to 26 or even 31 pounds the effectiveness did not approach that obtained with standard liquid lime-sulphur. The same lack of control is shown by the counts of the first-generation scales on the new wood.

## SUMMARY

The dry barium-sulphurs tested failed to show a satisfactory control of the San Jose scale at any of the strengths used. In a general way this material was about as effective as the other dry substitutes that were tested.

## GENERAL SUMMARY

The experiments here considered include 39 tests with dry calcium-sulphur, 13 tests with dry sodium-sulphur, and 11 tests with dry barium-sulphur. They were conducted under practical orchard conditions in four different States and cover a period of three years. Both apple and peach trees were used and conditions varied from a moderate infestation to incrustation. Conditions in the different orchards were such that the natural increase of the scale varied from very rapid to moderate.

Under the conditions mentioned the results obtained are sufficient to warrant the drawing of a general conclusion, at least for the localities in which these experiments were made.

The foregoing tests show that the commercial samples of dry calcium, sodium, and barium sulphurs, even when used at strengths much greater than ordinarily employed, do not furnish a satisfactory control of the San Jose scale.

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